

# PATENT SPECIFICATION

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## (54) DEVICE FOR DISSEMINATING VAPORIZABLE MATERIALS

(71) We, ALBANY INTERNATIONAL CORP., 1000 Providence Highway, Dedham, Massachusetts, United States of America, a Corporation organised and existing under the laws of the State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to device for disseminating vaporizable materials.

While the invention is primarily concerned with the practical methods of insect control, nevertheless the concepts here involved are applicable to all uses of controlled vapour dispersion. However, since insect attractants are of major importance, the description of the invention will be directed to such a usage.

That insects can be attracted or repelled by certain substances has been known for a great many years. In recent years, efforts have been made to utilize and extend this primitive knowledge for the purpose of controlling insect pests and mitigating the harmful effects they can inflict on man, animals, agricultural crops, and clothing. This has led to the recognition that olfaction plays a key role in communication among insects and that the media of communication are chemical substances produced and emitted by insects for communicative purposes. These chemical messengers have come to be called pheromones and are known to be highly specific as to insect species and elicited response. Pheromones may serve as alarm signals, food finding aids, mating signals, trail markers or defensive agents for warding off predators.

A great many insect sex attractant pheromones have been isolated and identified as to chemical composition and structure. They typically are straight chain or cyclic organic compounds of carbon, hydrogen, and oxygen, falling in the molecular weight range of 150 to 130. The structure and biological activity of most

of the known insect sex pheromones are extensively reviewed in the book, Insect Sex Pheromones, by Martin Jacobson, Academic Press, New York, 1972. Of the identified insect sex pheromones, many have been synthesized and the synthetic materials used in a variety of ways to facilitate insect pest control.

Two general methods of employing insect sex pheromones have been applied. One method involves using the pheromone to attract a target insect to a trap or to a point where it can be destroyed by an insecticide. Trapping also serves as a survey means of timing the application of chemical insecticides. A second method involves broadcasting small point sources of pheromone over an infested area to disorient the insects and make it difficult or impossible for the opposite sexes to find one another for mating. This latter method is referred to as the disruption technique, and is intended to suppress the pest insect population by subverting or interrupting the natural mating and reproduction process.

Effective and economical use of insect sex pheromones for accomplishing insect pest control, by whatever strategy, requires a suitable means of dissemination. A dissemination method or device must accomplish discharge of the pheromone to the atmosphere at a specific rate peculiar to the target insect, and for whatever period of time the particular adult insects are active in mating. Since synthetic insect sex pheromones are frequently rather expensive materials, the dissemination means or devices must be as efficient as possible in utilization of pheromone. Thus, a practical and economical sustained release system must be employed for disseminating pheromone which provides for a controlled discharge of the attractant at a specified rate and for a specified period of time.

Numerous examples of sustained or controlled release systems exist in the prior art of insect pest control. United States Patents 2,956,073; 3,116,201 and 3,318,769 teach the manufacture and use of

insecticides formulated into shaped plastic articles which serve to release the insecticide at a prescribed rate over an extended period of time. U.S. Patent 3,539,465 teaches the microencapsulation of hydrophilic liquid-in-oil emulsions in which polymeric capsular walls serve to mediate the controlled release of encapsulants such as insecticides and other biocidal agents. U.S. Patent 3,740,419 teaches the use of insecticide impregnated wood chips as a slow release pest control device. U.S. Patent 3,577,515 teaches the manufacture of microencapsulated insecticidal compositions by using interfacial polymerization to form a porous capsule wall which serves to regulate the rate of delivery of insecticide. U.S. Patent 3,590,118 describes a slow release insect repellent system in the form of a breathable acrylic film. U.S. Patent 3,592,910 discloses use of terpenoid resin-insecticide formulations which are designed to extend the period of effectiveness for nonpersistent or moderately persistent insecticides.

Each of these methods has its own particular cluster of advantages and drawbacks, which need not be elaborated here. Rather, it would be more in order to point out that researchers and economic entomologists continue to seek a more satisfactory scheme for the uniform, quantitatively predictable, prolonged automatic dissemination of miniscule amounts of active volatiles at an extremely low controlled rate. Sometimes the dissemination is desired on a tree-by-tree basis as, for example, in the control of certain orchard pests. Sometimes the dissemination is to be uniform and cover large areas, as in the use of pheromones to disrupt mating signals of insect pests attacking field crops. One possible superior means of achieving these ends entails the use of fine capillary tubes, both as containers and discharge devices.

Previous users of micro-tubes for pheromone delivery have employed them as containers and dispensers with both ends of the tube open to the atmosphere. The rate of pheromone discharged from the ends of the micro-tubes or micro-capillaries when used this way has generally been excessively high for many practical field applications. Moreover, when open at both ends such devices are subject to high material losses owing to for example mechanical shock, vibration and wind.

Our invention which comprises the use of microtubes or capillary channels of microconduits sealed at one end, while exceedingly simple, eliminates several significant obstacles to the use of such devices and confers on them practicability to an extent not heretofore contemplated by those familiar with the art.

As an example of the prior art, reference may be drawn to the article, "Novel Trapping and Delivery Systems for Airborne Insect Pheromones," by Lloyd E. Browne, et al., *J. Insect Physiol.*, 1974, Vol. 20, pp. 183 to 193. On pages 187—188, a laboratory testing scheme for assaying pheromone efficiency is described where the active liquid is filled into a 5  $\mu$ l glass capillary mounted vertically and open at both ends. The contained liquid is continuously evaporated from the liquid-air interface exposed at the bottom of the capillary to which it continuously feeds by the action of gravity. While the delivery rate may be kept quite constant, it is also quite high, being of the order of 1  $\mu$ l/minute. The dimension of the capillary tube is about 5 cm long by .4 mm diameter. A full charge of hexane will be discharged in about 5 minutes in the test described.

Another example of the prior art using micro-tubes is described by Shorey et al., in "Sex Pheromones of Lepidoptera. XXX. Disruption of Sex Pheromone Communications in 'Trichoplasia Ni' As a Possible Means of Mating Control," *Environmental Entomology*, Vol. 1, No. 5, October 1972, p.p. 641—645. Schemes for evaporation of pheromones at lower intermediate, and higher rates are discussed. It should be noted that in this work the authors view the use of micro-tubes as part of the higher rate technique.

"The substrates for higher evaporation rates were based on the principle of a liquid film of the pure chemical being exposed to the air. The rate was varied by varying the area of exposed film. These evaporators could be left in the field for several days without a decrease in their release rate. In practice, however, they were serviced and recharged daily. The 10 ng/min evaporator consisted of an 0.38 mm ID Teflon (Reg. Trade Mark) tube, 20 mm long, held vertically in a clip attached to the wooden stake. Loop lure was held in the lower end of the tube by capillarity. An inverted aluminum weighing cup attached to the top of the stake was used to shield the tube from excessive winds, which at times forced the loop lure out of unshielded tubes. The 30 ng/min evaporator consisted of 3 similar Teflon tubes held in a single clip".

The dissemination principle of this scheme is the same as that from the previous citation; liquid continuously evaporates at a liquid-air interface maintained by gravity at the bottom of a double open-end vertical capillary tube. The particular dissemination rates for those materials are at least an order of magnitude higher and up to 3 orders of magnitude higher than can be achieved by the use of our invention. It will also be noted that because of the double open-end configuration, the tube contents are unstably

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retained and subject to being blown out by wind. Note, also, the requirement for daily attention. Both of these objectionable situations are obviated by our device.

5 The present invention consists in a device for dissemination of a vaporisable material at a predetermined rate by vapour diffusion through a stagnant gas layer, comprising an elongated capillary conduit of  
10 predetermined cross-sectional area and length having one closed end and a vaporisable substance contained therein.

The invention also includes a method of dissemination of an insect attractant or  
15 insecticide material into the atmosphere wherein said material is contained in a device comprising a capillary conduit having one closed end and said material is disseminated at a predetermined rate by vapour diffusion  
20 through a stagnant vapour layer contained in said conduit.

The device according to the invention can be arranged to be easily loaded with a proper amount of material to be vaporised.

25 The device of the invention can be arranged to accomplish discharge of the vapour, particular a pheromone, to the

atmosphere at a specific rate peculiar to the target insect, and for whatever period of time the particular adult insects are active in mating. Thus, the dissemination of the substance to be vapourised is efficient in its utilization of the substance.

The device of the invention may be used to add to an environment, at a controlled rate, a vapour such as fragrances of flowers, fruits, or woods, for aesthetic effects. Another use for the device is for disseminating vapours having therapeutic uses, such as anti-histamine inhalants and biocides. Insect repellancy is also contemplated as a use for the device.

As evidence of the significantly improved controlled release capabilities of our invention, some data has been collected from the prior art and are present in Table I(A), below. Similar experiments performed by the present inventors involving the use of micro-tubes of polyethylene terephthalate open at both ends disseminating one of the pheromones identified in Part (A) is presented in Table I(B). By way of comparison, illustrations of the present invention are shown in Table I(C).

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TABLE I  
COMPARATIVE DISSEMINATION RATES  
Tube

Source	Evaporant	Dia (mm)	A (mm <sup>2</sup> )	Per Tube mg/hr	Rate Normalized mg/mm <sup>2</sup> /hr
(A) PRIOR ART - OPEN BOTH END TUBES					
1. Browne, J. Ins. Phys.	hexane	.4	.125	60	480
2. Shorey, Env. Ent.	loopleure	.38	.125	.0006	.0048
3. Pitman/Vité, Boyce Thompson	frontalin	.4	.125	5	40
(B) OUR WORK - OPEN BOTH END TUBES					
1. Vertical	frontalin	.16	.02	.12	6
2. Horizontal	frontalin	.4	.125	.89	7.1
3. Vertical	frontalin	.4	.125	1.3	10
(C) METHOD OF THIS INVENTION					
1.	frontalin	.4	.125	0.0068	0.054
2.	frontalin	.16	.02	.0009	.045
3.	CCl <sub>4</sub>	.2	.03	.018	.6
4.	o-dichlorobenzene	.2	.03	.002	.066
5.	cis-7-dodecenyl acetate	.2	.03	.000012	.0004
6.	cis-8-dodecenyl acetate	.16	.02	.000012	.0005
7.	linalool	.2	.03	.0003	.010
8.	disparlure	.2	.03	.00006	.002
9.	grandlure	.2	.03	.0003	.010

5 It is seen from I(A) that the dissemination  
rate for frontalin normalized to the hollow  
cross-section of the fibre is 40 mg/mm<sup>2</sup>/hr in  
the work by Pitman and Vité using glass  
capillaries 0.4 mm in diameter held  
10 vertically. This should be compared with our  
work using the same chemical in 0.4 mm PET  
tube open at both ends held vertically (Table  
I(B)). The difference between 40 mg/mm<sup>2</sup>/hr  
15 and 10 mg/mm<sup>2</sup>/hr can be accounted for in  
that the method of Pitman and Vité involves  
the use of an olfactometer with a controlled  
forced velocity of air across the open end of  
the tube, whereas in our experiment the  
20 evaporation was carried out in a laboratory  
with virtually stagnant air. The evaporation  
in both cases occurs at the air-liquid  
interface continuously presented at the  
bottom of the micro-tubes. In our  
25 experiment, the same micro-tube held  
horizontally results in a dissemination rate of  
7.1 mg/mm<sup>2</sup>/hr, somewhat lower than when  
held vertically. A smaller (0.16 mm) capillary  
when held vertically exhibits the slightly  
30 lower rate of 6 mg/mm<sup>2</sup>/hr, but still quite  
comparable to that experienced with the  
larger capillary. The important point to be  
noted is that all these tests lie in the same  
order of magnitude of dissemination level,  
i.e., 10 mg/mm<sup>2</sup>/hr.

By contrast now with the material in Table  
I, Parts (A) and (B), there are data presented  
in Table I(C) on the controlled prolonged  
rate of dissemination exhibited by the  
35 devices of our invention. The first two lines  
in I(C) present information on the  
dissemination of frontalin from two different

sizes of micro-tube. It will be seen that these  
two rates are quite consistent with one  
another, being .054 and .045 mg/mm<sup>2</sup>/hr. 40  
These values differ by a factor of 200x from  
the data in I(B), and by as much as 1,000x  
from the data in I(A). Lines 3 and 4 of I(C)  
present evaporation rates for two very  
common materials, carbon tetrachloride and 45  
ortho-dichlorobenzene, one of high and the  
other of moderate volatility. It is seen that  
their dissemination rates are in the  
proportion of 10:1 relative to one another,  
quite in keeping with their relative vapour 50  
pressures at room temperature. However, by  
contrast with the dissemination of hexane  
(Line 1 of Table I(A)), with a volatility  
comparable to carbon tetrachloride, they are  
in the order of 1,000 to 10,000 times less 55  
rapidly evaporated. The remainder of Table  
i(C) describes dissemination rates for a  
number of pheromones. Line 5, describing  
the dissemination of looplure (cis-7-  
dodecenyl acetate), should be contrasted 60  
with the work of Shorey shown on the second  
line of I(A). Here it will be seen that there is a  
ten-fold reduction in dissemination rate by  
our invention. Line 6 data describing cis-8-  
dodecenyl acetate is quite consistent with 65  
the dissemination data for the cis-7 isomer.  
The data of lines 7, 8, and 9 are further  
confirmation of the capabilities of our  
invention.

Table II provides the chemical 70  
descriptions and insect targets for several of  
the selected pheromones for which data is  
presented in Table I.

TABLE II  
CORRELATION OF COMMON NAME, CHEMICAL NAME, AND TARGET INSECT  
OF EVAPORANT FROM COMPARATIVE RATES TABLE (i.e., TABLE I)

Common Name (Evaporant)	Chemical Name	Target Insect
Carbon tetrachloride	Tetrachloromethane	-
o-dichlorobenzene	1,2-dichlorobenzene	-
Disparlure	Cis-7,8-epoxy-2-methyl octadecane	Gypsy Moth
Cis-8-dodecenyl acetate	Cis-8-dodecenyl acetate	Oriental Fruit Moth
Frontalin	1,5-dimethyl-6,8-dioxabicyclo(3,2,1) octane	Pine Bark Beetle
Grandlure Compounds I-IV:		Boll Weevil
Compound I:	Cis-2-isopropenyl-1-methylcyclobutaneethanol	
Compound II	Cis-3,3-dimethyl- $\Delta^1, \beta$ -cyclohexaneethanol	
Compound III	Cis-3,3-dimethyl- $\Delta^1, \alpha$ -cyclohexaneethanol	
Compound IV	Trans-3,3-dimethyl- $\Delta^1, \alpha$ -cyclohexaneethanol	
Hexane	Hexane	-
Linalool	3,7-dimethyl-1,6-octadien-3-ol	-
Looplure	Cis-7-dodecenyl acetate	Cabbage Looper

Figure 1 filled tubes mounted on a base in upright position.

Figure 3a illustrates a method of assembling a continuous parallel array of filled hollow fibres on an adhesive backing tape, with periodic melt-sealed zones

occluding the tubes, and

Embodiments of the invention will now be described, by way of example, with reference to the accompanying drawings, in which:—

Figure 1 is an elevation in section, showing a representative filamentary tube of this invention filled with a vaporizing material.

Figure 2 illustrates a group or stack of the

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Figure 3b illustrates how a single dispenser can be cut from such a tape.

Figure 4a illustrates a single hollow fibre filled with evaporant, open at one end and self-sealed shut at the other by melt bonding:

Figure 4b illustrates the same configuration of hollow fibre where the fibre has not been severed through the heat-sealed region but consists of two microconduits each with one open end and one closed end.

Figure 5 is a graph illustrating the release rate of carbon tetrachloride using this invention, being Example 3 of Table I(C) given previously.

Figure 6 is a graph illustrating the release rate of *o*-dichlorobenzene using this invention, being Example 4 of Table I(C) given previously.

Figure 7 is a graph illustrating the release rate of linalool using this invention, being Example 7 of Table I(C) given previously.

Figure 8 is a graph illustrating the curve of release rate for disparlure, using this invention, being Example 8 of Table I(C) given previously.

Figure 9 is a graph illustrating the curve of release rate for grandlure, using this invention, being Example 9 of Table I(C) given previously.

Figure 10 is a plot of release vs. time for frontalinal disseminated from a double-open end tube, being Example 3 in Table I(B) presented previously.

Figure 11 is a plot of release vs. time for frontalinal disseminated according to this invention, being Example 1 of Table I(C) presented previously.

Figure 12 is another embodiment of the invention in which, instead of using separate tubes, flat sheets are embossed to form channels (rectangular channels being shown, although they could be other shapes, such as semi-circular) and this sheet is adhered to a base sheet in order to provide a plurality of parallel capillary tubes. The channels in this illustration are unfilled and unsealed.

Figure 13 illustrates transverse sealing zones imposed on the parallel array of channels in Figure 12, after filling same with latent evaporant.

Figure 14 is another view of the device of Figure 3, in which a structure is shown having the tubes fastened to a material such as an adhesive coated backing tape, and with the tubes heat sealed to close them at regular intervals along the length of the structure.

Figure 15 is an end view, enlarged, of the Figure 14 structure.

Figure 16 is a view taken of the Figure 14 structure, in the direction of sight lines 16—16, the view being in section.

Throughout the drawings, similar reference characters indicate corresponding parts. Dimensions of certain of the parts as shown in the drawings may have been

modified and/or exaggerated for the purpose of clarity of illustration and understanding of the invention.

This invention contemplates as a preferred embodiment the use of hollow fibres as reservoirs and dispensers for the vapours to be disseminated for several purposes (such as fragrances for artificial flowers), but particularly for insect sex pheromones where pheromones are employed as trap baits or as a means of interrupting natural insect mating processes by the so-called disruption technique. Pheromone deposited in the core of hollow filaments of appropriate length and internal diameter is released by evaporation from one end of a small tube, the other end being sealed.

The hollow filaments of the invention may be made from any one of a number of natural or synthetic polymeric materials by any of the processes commonly employed in producing man-made fibres. Useful materials include polyesters, polyolefins, acrylics, modacrylics and polyamides. The selection of an appropriate material will be governed by considerations of chemical compatibility or inertness of the fibre material with the chemical agent or formulation to be incorporated in and released from the hollow filament. Where broadcast distribution of the pheromone hollow filaments is contemplated, such as when employing the disruption technique, environmental considerations might dictate the use of a biodegradable fibre material. Regenerated protein or cellulosic fibre materials would satisfy such a requirement.

Referring now to the drawings, as illustrated in Figure 1, a capillary tubular filament 2 of this invention, which has a bore or lumen 4, is closed at one end by means such as heat-sealing or a plug 6 such as epoxy cement or other suitable material, and is loaded with the insect attractant 8. The attractant 8 has preferably a wetting type meniscus 10 at its open or upper end in respect to the tube material. If such a meniscus is not obtained at between the vaporizable material and tube, then for a given vaporizable material the proper tube material is selected.

Referring to Figure 2, a group 12 of tubes 2 is shown these tubes being held together by conventional means (not shown), such as an exterior wrapping or by being cemented together or by being placed in a suitable outer container. The open ends of the tubes are at the top and the bases of the tubes are mounted by conventional means on a support or base 14. The total amount of insect attractant, or flower fragrance, where the invention is used to provide a fragrance for artificial flowers, that will be released, will depend, as set forth above and below, on the size of the tubes, the particular material

used, and the number of tubes which make up the group 12. Since there is an extremely large number of possible variations of these factors, it is impossible to list all of the combinations nor is it necessary to understand and apply this invention. Persons skilled in the art can readily bundle the required number of tubes once the release rate of a given tube containing a given material is known. As was illustrated in Table I(C), for exemplary insect attractants, the release rate is readily determinable. Therefore, all that is needed to design a disseminator for a given total quantity of material is to take the number of hours of release desired, the weight of the insect attractant needed for that number of hours; the amount of attractant per tube will be known by simple computation and then the number of such tubes which need to be placed together to form a group 12 can be readily computed (i.e. by dividing the attractant amount per tube into the total amount).

Referring to Figure 3a and Figure 3b, another embodiment of the invention is shown, where a group of capillary tubes like tubes 2 are adhered to a backing tape, periodically sealed along their length, and rolled up in the form of a dispensing tape as shown at 15. The insect attractant 8 does not escape from the tubes until the tubes are severed at selected spots between seal regions. Further detail on this method is given by Figures 14, 15 and 16.

The above two embodiments illustrate two ways of mounting the tubes, and the groupings of the tubes can be large or small, depending upon the particular requirement. In actual use, around a field of plant life which it is desired to protect from insects, a number of the structures of either Figure 2 or Figure 3 can be placed, so that regardless of which way the wind blows, insects will be attracted to at least some of the sites. Generally, these embodiments will be used in configuration with insect traps for the monitoring of pest populations or their elimination by direct trapping. Figure 4 illustrates another embodiment wherein single filled fibres are sealed and cut to expose one or two closed-end channels. These single fibres may be dispersed broadcast over crops or fields from suitable ground-based or airborne dispensers and may be used for the disruption method of pheromone application.

Referring to figure 5, there is shown a typical release rate curve (plotted in terms of weight loss as the ordinate against time as the abscissa) of carbon tetrachloride which is used as an exemplary material in order to establish a model release rate. It will be observed that at the very start, the weight loss is relatively high, as indicated by that

portion of the curve A which falls steeply shortly after the start of the release. The curve then flattens out and becomes almost horizontal as indicated at the portion B. It is this prolonged low level of release that comprises one of the most important virtues of our invention. The exact shape of the curve varies somewhat, depending upon the material used and the size of the tubes, but in general it can be said that the curves of all materials tested fall in a similar class of curves.

Referring to Figures 6, 7, 8 and 9, curves are shown for the use of the invention, respectively, for *o*-dichlorobenzene, linalool, disparlure, and grandlure, respectively. Their compositions or chemical name are as follows:—

- linalool—terpene alcohol
- disparlure—cis-7,8,-epoxy-2-methylocta-  
decane
- grandlure—a mixture consisting of:  
(a) cis-2-isopropenyl-1-methylcyclobutyl-  
ethanol
- (b) cis-3,3-dimethyl cyclohexyliden-  
ethanol
- (c) cis-3,3-dimethyl cyclohexylidene-  
acetaldehyde
- (d) trans-3,3-dimethyl cyclohexylidene-  
acetaldehyde

Where controlled release of more than a single substance is desired, it is possible to employ bundles or groups of individual fibres charged with different volatile materials. By appropriate selection of individual fibre diameters or the relative numbers of fibres charged with different materials, vapour mixtures can be disseminated with controlled composition. Examples of this variant are (1) the combined use of attractants and toxicants, (2) attractants which are chemical mixtures or which require a chemical synergist in precise ratios in order to be effective, and (3) fragrances or deodorants requiring a mixture of chemicals for optimum effect.

This variant is particularly useful where mixtures of chemicals of substantially different volatilities must be dispensed to give a vapour mixture of rather constant composition. Appropriate selection of fibre lengths, as well as diameter or number, allows one to compensate for different rates of evaporation, thereby delivering a vapour mixture of controlled and constant composition. The variant is also useful when the materials one desires to release simultaneously at a controlled rate are incompatible with one another (i.e. immiscible or chemically reactive) in the condensed phase.

As indicated above, the purpose of having tubes of different sizes is that the variation in

the diameters of the tubes is one of the factors that controls the rate of evaporation or release rate of the attractant. Instead of using tubes of various diameters, an alternative is to use tubes of the same size but use more tubes filled with one material than the number of tubes filled with another material, the ratio of tubes determining the resultant vapour compound ratio. If, for example, in a vapour dispersion of two compounds, it is desired to have three parts of one compound to one part of the other compound as the vapours mix on emerging from the tube ends, then a bundle of tubes of a predetermined like diameter would have three times as many tubes of one compound as there are tubes of the other compound, thus giving the required compound mixture. Of course, in obtaining a vapour which is a mixture, the evaporation rates of the individual vaporizable materials need to be considered as factors, and evaporation rates can be usefully employed together with tube diameters, or ratios of tubes of one material to tubes of other materials, to obtain the desired vapour. The combinations of such variables are almost infinite in number and therefore are not set forth herein. It is possible by this technique and by this invention to produce vapours comprising a plurality of discrete compounds. In actual practice, what the manufacturer of the device could do would be to preload tubes of various materials; and then on a custom order basis, the requisite number of tubes would be bundles in accordance with the customer's desires as to the relative proportions of the emergent vapour. In compounding the tube bundles, of course, the weight loss curves for the several compounds would be taken into consideration.

As an example, if the customer desires an attractant together with an insecticide the vapour of which is the killing factor, the customer would order tubes some of which in the bundle would contain the attractant and the other would contain the insecticide liquid. The vapours mix as they emerge from the tubes to produce a mixture of the desired amount of insecticide and attractant. The attractant would attract the insects to the site where the bundled tubes would be stationed, and upon the insects going to the area of the vapours, the insecticide would kill them.

Figure 10 is a plot of release rate vs. time for frontal dissemmination according to prior art methods from a 0.4 mm PET micro-tube, being the data of Line 3 of Table I(B). It illustrates the high constant rate characterizing this method.

Figure 11 illustrates dissemmination of the same pheromone from the same tube as used in Figure 10, except as defined by the present invention. It clearly shows the significantly

lower controlled rate achieved after the initial rapid fall in rate as described for the previous examples of this invention. (Note that the abscissa in Figure 11 is in  $\text{gms/hr} \times 10^5$  while that in Figure 10 is in  $\text{gms/minute} \times 10^5$ ).

Referring now to Figure 12, an embodiment is shown in which the capillary tubing is provided by first forming a sheet indicated generally by numeral 40 in such a manner that it has the channels 42 therein. These channels are shown as five in number, but any number desired could be utilized. Also, channels 42 are shown as being rectangular in cross-sectional configuration, but semi-circular, oval, or other polygonal channels could also be used. It will be noted that the bottoms of the channels are open, and the sheet 40 is then adhered to a base sheet 41 by attaching the webs 46 securely to the base sheet 44 using conventional adhesives. As a result, between the base sheet 44 and the channel members 42 are provided the capillary tubings 48 as shown, of predetermined cross-sectional shape and size.

Figure 13 illustrates the composite sheet channel system of Figure 12 filled and sealed across the conduits, as at 49.

Referring now to Figures 14, 15, and 16, more detail is shown of the embodiment of the invention illustrated by Figures 3a and 3b. An elongated strip 64 of suitable material having an adhesive coating 66 thereon is utilized. Such a material can be conventional masking or wrapping tape which is coated with a pressure-sensitive adhesive. A plurality of filamentary tubes 68 is stretched lengthwise along the combined tape 64, 66 these filamentary tubings being held to the tape by means of the adhesive 66. If it is desired, the adhesive 66 can be of the kind which prior to use is coated with a protective pull-off cover, but in use when the cover is removed and the tubes 68 are adhered thereto, the adhesive then sets in air in order to form a strong fastening of the tubes 68 to the strip 64.

The tubes 68 may be placed upon the strip 64 before or after being filled. After they have been filled and are mounted upon the strip, they are heat sealed along the junction lines 70, the tubing material being of a kind which can be heat sealed by the application of a heated sealing member. By so heat sealing, the tubing wall is collapsed at the points 70 thus providing for each elongated tubing a plurality of sections 72, each section 72 being but a portion of a total length of the individual filaments. In use, the user will cut across the tubings in order to provide short conduit elements of the desired length of tubing, the cutting taking place between the heat sealed portions 70. The length of tubing sections from the closed heat sealed end to the open end, where the cutting takes place,

will determine, other parameters being considered in accordance with the teaching of this invention, upon the actual length of the individual section 72. The Figures 14—16 embodiment give a convenient way in which to supply to a prospective user either a roll or a flat length of filamentary tubes, already mounted on a base, but in which the tubes are sealed against any loss of material until it is desired to use the tubes. When the user desires to use a certain portion, he will take the material and from an end he will then cut the length of tubing (the cutting taking place across the width of the strip) in order to open the ends of the tubing. The length of the tubing which he determines to cut will depend (other factors can also be considered) on the number of hours he wishes the cut length of tubings to emit their vapours.

In the practice of this invention, the tubular fibres of polymeric material fabricated to convenient dimensions are loaded with an insect attractant by one of the methods described below. The hollow fibre dimensions for practical consideration are generally in the range of approximately 0.025 to 1.0 millimeters in external diameter, and 0.01 to 0.8 millimeters in internal diameter, although it will be obvious to one familiar with the art of fibre extrusion that microtubes both larger and smaller than these limits are attainable and may be considered variants of this invention. (In the ensuing description, the conventional abbreviations of the metric system will be used, for example, mm for millimeters, cm for centimeters and g for grams). The illustrated dimensions represent, therefore, a preferred rather than a limiting range. Hollow fibre length will be governed by the length of time desired for release of the attracting agent. Thus, for a given attractant material, the invention allows control over release rate through the number and lumen diameter of fibres employed and control over the period of activity through selection of appropriate fibre length. Release rate curves describing the character of release behavior for this invention typically show a brief period of high release rate followed by a long period of somewhat asymptotic behavior, where the release rate decline, as reflected in the slope of the "asymptotic" portion of the curve, is so small as to approximate a linear release rate. Such release rate curves are given below in respect to several of the examples of the invention that have been made and tested successfully.

The following examples will serve to illustrate the practice and utility of this invention.

#### Examples

##### Example 1.

This example was given as Line 3, Table I(C) and describes the release or evaporation behavior of carbon tetrachloride (a model compound for relatively volatile attractants or insecticides), from undrawn hollow fibres of polyethylene terephthalate. Fibre dimensions were 0.254 mm external diameter and 0.203 mm internal diameter. Hollow fibre lengths of 127.0 mm to 203.2 mm were loaded with carbon tetrachloride, sealed at one end with an epoxy potting compound, and mounted in a vertical position, open end up, on a flat surface. The lumen cross-sectional area was approximately  $3.245 \times 10^{-4} \text{ cm}^2$ . Loss of carbon tetrachloride from the fibre core by evaporation and diffusion out of the open end of the fibre was measured by following the liquid meniscus recession into the fibre interior by means of a cathetometer. Incremental volume losses converted to weight losses and plotted as a function of time are displayed in Figure 5. In this case, a quasilinear release rate is observed after 30 hours.

The release was measured at quasi-steady rate of weight loss, and the figure given is the average of five samples each.

##### Example 2.

O-dichlorobenzene, serving as a model for insect attractants or insecticides of intermediate volatility, was loaded by a capillary filling technique into an undrawn polyethylene terephthalate hollow fibre with lumen cross-sectional areas of  $3.09 \times 10^{-4} \text{ cm}^2$ . Filled hollow fibres 127 mm in length were sealed at one end with an epoxy potting compound, and mounted in a vertical position, open end up, on a flat surface. Release of the o-dichlorobenzene from the hollow fibres was observed and measured by following meniscus recession into the fibre interior. Figure 6 displays the release curve for this experiment, the data having previously been given as Line 4 of Table I(C). The quasilinear release rate was reached at about 90 hours.

##### Example 3.

The data for this Example was Line 7 of Table I(C). The terpene alcohol, linalool, chosen as a model for grandlure, the aggregating pheromone of the cotton boll weevil, was loaded into undrawn polyethylene terephthalate hollow fibres with a lumen cross-sectional area of  $3.14 \times 10^{-4} \text{ cm}^2$ . 102 mm to 127 mm lengths of loaded fibre, sealed at one end with a potting compound, were mounted in a vertical

position, open end up, on a flat surface. Release of the linalool from the hollow fibres was monitored by following meniscus recession into the fibre with a cathetometer. Figure 7 is the release curve for linalool. After about 40 hours, the release rate becomes steady at  $5 \times 10^{-6}$  g/min. Assuming a desired release rate for a boll weevil bait of  $3 \times 10^{-4}$  g/day, and a required attraction period of 168 days, a bait configuration is calculated to require 42 fibre open ends with individual fibre lengths of 3.8 cm.

#### Example 4.

This data was given on Line 8 of Table I(C). Disparlure, the sex attractant for male gypsy moths, was loaded into undrawn polyethylene terephthalate hollow fibre with a lumen cross-sectional area of  $3.4 \times 10^{-4}$  cm<sup>2</sup>. The disparlure release curve was obtained as described in Example 1. The release rate became steady at  $1.44 \times 10^{-6}$  g/day/fibre end as from the release curve displayed in Figure 8. The desired release rate for a gypsy moth sex pheromone trap bait is  $2.16 \times 10^{-4}$  g/day and the desired period of activity is 90 day. Thus a hollow fibre trap bait would require three fibre open ends and an individual fibre length of 0.46 cm per open end.

A commercial insect trap lined with a sticky substance to hold attracted moths in the trap was baited with disparlure loaded hollow fibres and placed in a wooded area of Norfolk County, Massachusetts, during the month of August, 1974. The pheromone baited trap captured male gypsy moths at a ratio of 3:1 over an unbaited trap.

#### Example 5.

This data was given as Line 9 of Table I(C). Grandlure, the aggregating pheromone attractant of the cotton boll weevil, was loaded into undrawn polyethylene terephthalate hollow fibre with a lumen cross-sectional area of  $3.14 \times 10^{-4}$  cm<sup>2</sup>. The grandlure release curve was obtained as described in Example 2. Release rate became steady at  $5 \times 10^{-6}$  g/min/fibre end, as shown in the release curve displayed in Figure 9. The desired release rate for a cotton boll weevil pheromone bait is  $3 \times 10^{-4}$  g/day and the desired period of activity is 168 days. Thus, a hollow fibre trap bait would require 40 fibre open ends with individual fibre lengths of 0.4 cm per open end.

The manner in which attractant materials are charged into the hollow filaments of this invention may be one of several. The liquid attractant formulation will fill the hollow filament by capillary action or by gravity feed using the filament as a siphon. (In the siphon method, one end of a tube or tubes is inserted in the desired liquid. The other ends of the tubes are below the liquid surface. A

slight suction is applied to the lower ends. Once the liquid flow has started, then the siphon action continues and the tubes fill). Another filling method involves simply sucking liquid into the fibre core by placing fibre ends beneath the liquid surface and evacuating the fibres from the other end by means of a suction bulb or aspirator device. Yet another involves placing fibre segments beneath the liquid surface and compressing them to force out air, after which they contract and draw the fluid. It is also possible to fill the fibre at the time of spinning by injecting the liquid attractant formulation as a core fluid during the spinning operation. Other methods of filling the filament capillary may come to mind, but it should be noted that the particular method of filling is not a part of this invention.

#### WHAT WE CLAIM IS:—

1. A device when used for the dissemination of a vaporisable material at a predetermined rate by vapour diffusion through a stagnant gas layer, comprising an elongated capillary conduit of predetermined cross-sectional area and length having one closed end and a vaporisable substance contained therein.
2. A device according to claim 1 wherein two said conduits are joined at their closed ends.
3. A device according to claim 1 or claim 2 wherein the device comprises a plurality of said conduits.
4. A device according to claim 3 wherein the said conduits are of at least two different cross-sectional areas.
5. A device according to claim 3 or 4 wherein each said conduit is closed at a predetermined interval along its length.
6. A device according to any one of claims 3, 4 or 5 wherein said conduits are formed by occluded corrugations in a sheet material.
7. A device according to any one of claims 3 to 6 wherein said conduits are attached to a common backing strip to form a composite structure.
8. A device according to claims 6 or 7 wherein the composite structure is wound up in a helical coil.
9. A device according to any one of the preceding claims wherein the conduits are formed from a polyester, a polyolefin, a polyamide, a polyacrylic or a polymodacrylic material.
10. A device according to any one of the preceding claims wherein the vaporisable material is an insecticide and/or an insect attractant, or a mixture thereof.
11. A device according to claim 10 wherein the insect attractant has a flower like odour.
12. A device according to claim 10 wherein the insect attractant is a pheromone.

13. A device according to any one of the preceding claims wherein the conduit is a hollow fibre.

5 14. A device according to any one of claims 3 to 9 wherein some of said conduits contain an insect attractant and others contain an insecticide.

10 15. A device according to claim 1 for the dissemination of a vaporisable materials substantially as herein described with reference to and as shown in the accompanying drawings.

15 16. A method of dissemination of an insect attractant or insecticide material into the atmosphere wherein said material is contained in a device comprising a capillary

conduit having one closed end and said material is disseminated at a predetermined rate by vapour diffusion through a stagnant vapour layer contained in said conduit.

17. A method of dissemination according to claim 1 of an insect attractant or insecticide material substantially as hereinbefore described.

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COMPLETE SPECIFICATION

5 SHEETS

This drawing is a reproduction of  
the Original on a reduced scale  
Sheet 1

FIG. 1

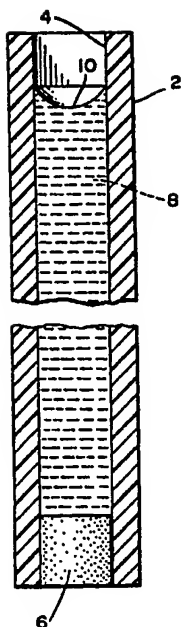
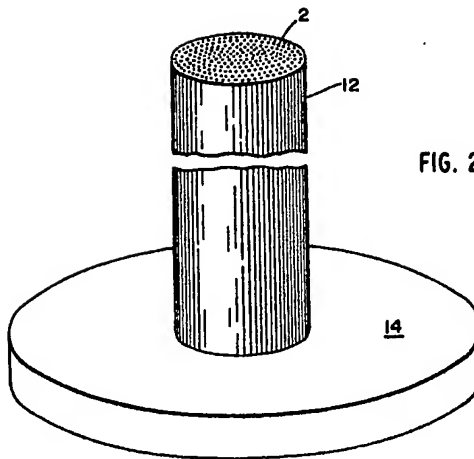


FIG. 2



CONTINUOUS TAPE  
FROM WHICH SINGLE  
DISPENSERS ARE CUT

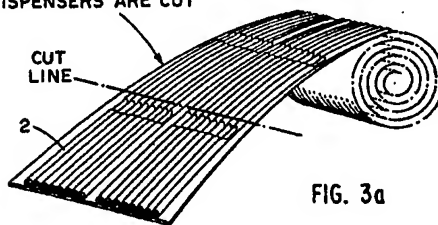


FIG. 3a



FIG. 4a

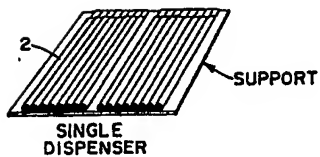


FIG. 3b

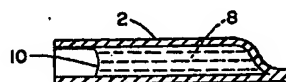


FIG. 4b

BEST AVAILABLE COPY

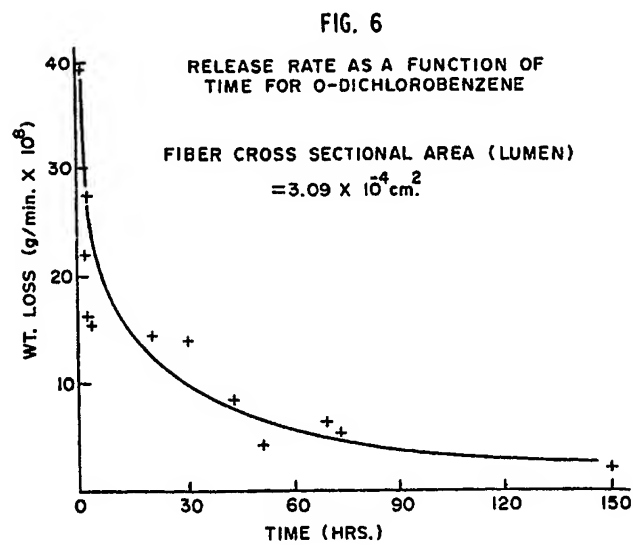
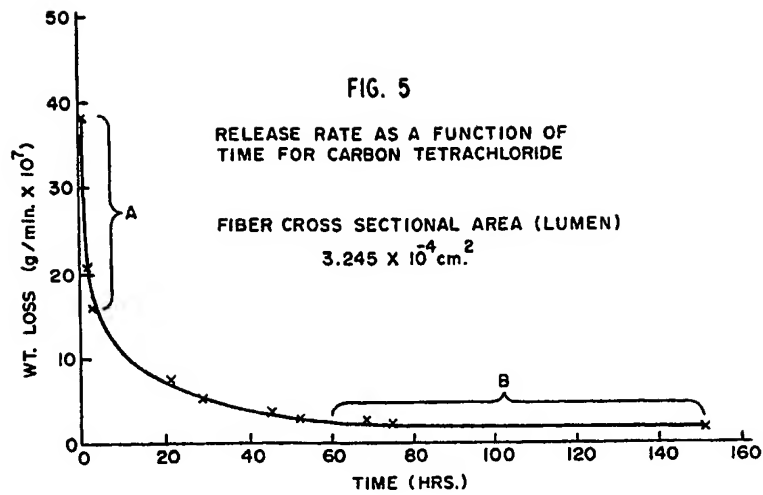


FIG. 7

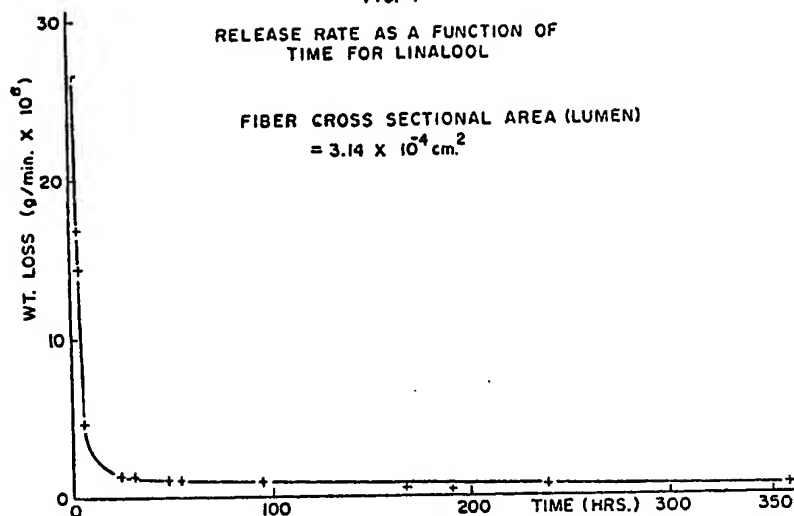
RELEASE RATE AS A FUNCTION OF  
TIME FOR LINALOOLFIBER CROSS SECTIONAL AREA (LUMEN)  
 $= 3.14 \times 10^{-4} \text{ cm.}^2$ 

FIG. 9

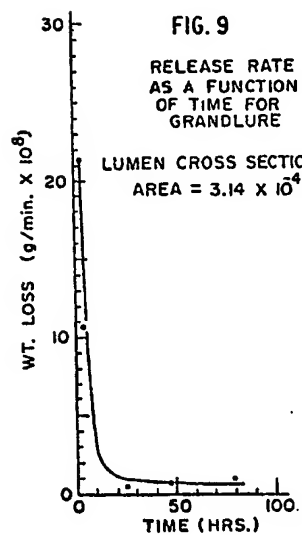
RELEASE RATE  
AS A FUNCTION  
OF TIME FOR  
GRANDLURELUMEN CROSS SECTIONAL  
AREA =  $3.14 \times 10^{-4} \text{ cm.}^2$ 

FIG. 8

RELEASE RATE  
OF DISPARLURE  
AT 70°F 65 RHFIBER AREA =  $3.14 \times 10^{-4} \text{ cm.}^2$   
(LUMEN)